CCQM Activities in the Organic Analysis Working Group

NIST's Analytical Chemistry Division has participated in 40 of 55 Consultative Committee for Amount of Substance – Metrology in Chemistry (CCQM) Organic Analysis Working Group (OAWG) studies since 1997, and coordinated a number of studies during the past year. The CCQM conducts international comparisons to establish equivalence among measurements made by national metrology institutes (NMIs). OAWG studies during the past two year included measurement of vitamins in infant/adult formula, hormones in human serum, volatile organic compounds in methanol, ethanol in water, and polycyclic aromatic hydrocarbons in soil and particulate matter.

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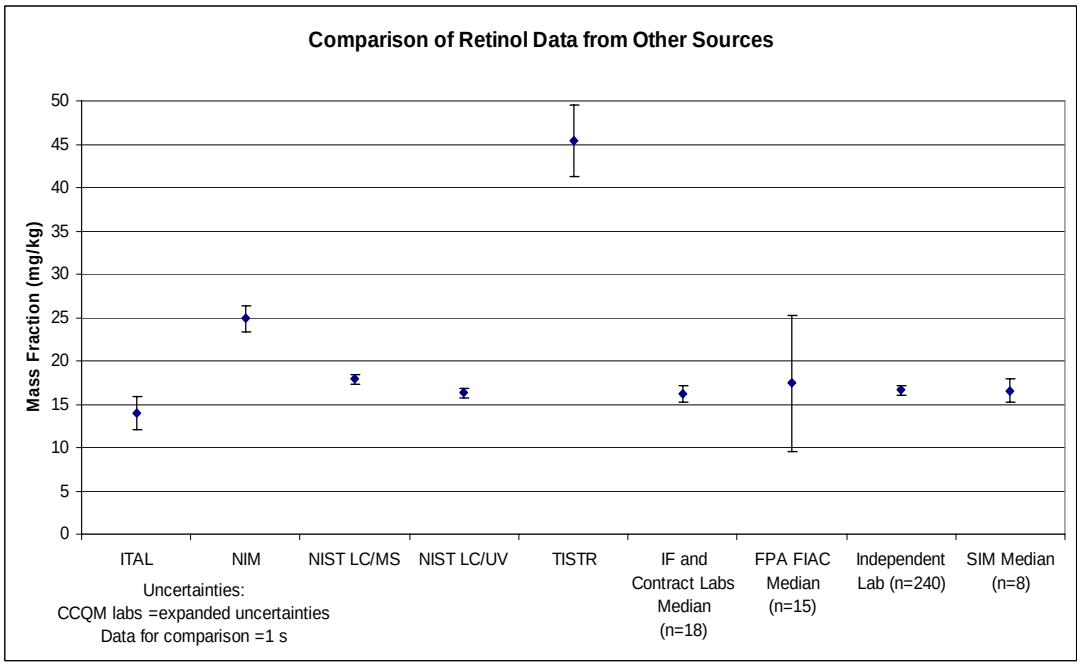
In past years, NIST has coordinated or participated in CCQM studies in areas related to the environment and clinical chemistry, including studies in which purity of various compounds was assessed. ACD coordinated the following CCQM studies within the OAWG during 2006: CCQM-P77a and b Cortisol and Progesterone in Serum, CCQM-P78 Nutrients in Adult/Infant Formula – the first food nutrition study, CCQM-K27.2 Ethanol in Water, and CCQM-K47 Volatile Organic Compounds (VOCs) in Solvent. A particulate matter sample that meets the homogeneity requirements for use in the planned CCQM-K50 for PAHs in soil and particulate matter was characterized.

Six NMIs or their designees participated in the pilot study CCQM-P77 Cortisol and Progesterone in Serum with one of the NMIs returning data for cortisol only. With the exception of one of the designated laboratories that used an immunoassay method, the data agreed well among the remaining five laboratories. For the cases were there were minor discrepancies, potential sources of error have been identified, and NMIs will investigate and report their findings in April 2007. A key comparison is planned for 2007.

Five NMIs or their designees participated in the pilot study CCQM-P78 Nutrients in Infant/Adult Formula, in which vitamin A, folic acid, and niacin were measured; one of the NMIs returned data for only one of the three analytes. Since the same sample had been used in other interlaboratory studies, the data among the NMIs could be compared even though a limted number of NMIs participated (see Figure). A key comparison is planned following the April 2007 OAWG meeting.

Key Comparison CCQM-K27.2 was the second subsequent study for Ethanol in Aqueous Matrix. Five laboratories participated, with the data from four of those laboratories agreeing within 2%. The data for the four laboratories are comparable to the data from the previous two key comparisons so this study will be finalized.

Key Comparison CCQM-K47 Volatile Organic Compounds in Solvent and the subsequent pilot study CCQM-P61.1 run concurrently were co-coordinated by NIST and CENAM in Mexico. There were five participants in CCQM-P61.1 and eight participants in CCQM-K47. Because there are discrepancies between the participant results and the gravimetric concentrations from the preparation of the solutions, the National Meterology Institute of the Netherlands also analyzed the study samples against their gas standards. The data confirmed the gravimetric concentrations for three of the four analytes of interest. Further discussions will be held at the April 2007 meeting to determine the Key Comparison Reference Values (KCRVs) for CCQM-K47.



Four NMIs or their designees participated in the pilot study CCQM-P68 19-Norandrosterone in Human Urine, which was coordinated by the National Measurement Institute of Australia (NMIA). In order to participate in this intercomparison, NIST developed a new LC/MS/MS method for 19-norandrosterone. The results from the four NMIs were in excellent agreement, with a relative standard deviation (RSD) of only 1.7%.

Impact: The results from these studies will be used to benchmark the appropriate Comparibility and Measurement Claims (CMCs) of the participating NMIs.

Future Plans: The materials identified for key comparison CCQM-K50 will be distributed early in 2007 for discuss at the October 2007 meeting. Key comparisons are planned for nutrients in infant formula and for cortisol and progesterone in serum.